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(54) **Clarification agent for coloured fabrics and method for treatment of fabrics.**

(57) The clarification agent contains as the active component a cellulase. Fabrics which due to use for extended periods of time and repeated washings have acquired a dull, greyish appearance can regain their original clean colours by treatment with the clarification agent in aqueous medium. The clarification agent is considered as being the first and only agent of this kind. During the method for treatment of textiles a pH-value not too far from the pH optimum of the cellulose in question is usually used.

Fig.8.

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This invention relates to a clarification agent for coloured fabrics and a method for treatment of fabrics. The fabrics comprised by the invention are cellulose based, e.g. fabrics manufactured of cotton, of synthetic fibres on cellulose basis, e.g. rayon, of flax, hemp, jute, or ramie, or of mixtures containing one or more of such fibres.

Clothes made from the above fabrics often develop a greyish appearance after having been used and washed repeatedly. This unwanted effect is particularly evident in case of dyed clothes, especially clothes with dark colours. This greyish appearance is probably caused by disordered fibres which are broken or torn up by mechanical action. Even after thorough wash in which ordinary dirt, such as protein, oil, starch and dust, has been removed, the clothes still look worn and fluffy.

Thus, a need exists for a clarification agent for coloured fabrics, containing or consisting of cellulose based fibres, which clarification agent can reestablish the attractive look of fabrics which have developed a greyish appearance, thereby offering the consumer a chance to avoid discarding clothes before it is actually needed.

Now, surprisingly, according to the invention it has been found that the attractive look of fabrics, containing or consisting of cellulose based fibres, which have developed a greyish appearance, can be reestablished, if the fabric is treated in an aqueous, cellulase containing medium.

Thus, the invention in its first aspect comprises a clarification agent for coloured fabrics, containing or consisting of cellulose based fibres, wherein the clarification agent as an active constituent contains a cellulase. It is to be understood that the cellulase can be used as such or in admixture with additives, e.g. salts, surfactants or formulated detergents. Also, it is to be understood that the cellulase can be any cellulase known to the art. Some microbially produced cellulases are preferred, as will appear from the following.

To the best of the knowledge of the applicants no one has previously solved this problem. Thus, our invention is not an invention of a new agent for a specific purpose, in relation to

which already some inferior agents belong to the prior art, but the very first invented agent known to fulfil this purpose, and furthermore an agent which fulfils the purpose excellently, which will appear from the documentation presented in the following.

The invention as explained above is related to coloured fabrics, i.e. fabrics with another colour than white. However, the agent according to the invention will remove the disordered fibres on a white fabric as well, but this action will hardly be visible to the naked eye as it is in relation to fabrics with another colour than white.

It appears from GB patent No. 1 368 599 that cellulases can be used as a harshness reducing agent for harsh fabrics, and also, from US patent No. 4 435 307 it appears that a special cellulase, i.e. the Humicola insolens cellulase, can be used for this purpose. It is to be understood, however, that the harshness reducing effect and the colour clarification effect are two different effects, inasmuch as the former is needed in relation to fabrics with a stiff feeling and the latter is needed in relation to fabrics with a greyish appearance. Thus, in relation to a wanted improvement of a fabric with a greyish appearance no guidance to perform the improvement can be derived from the knowledge that cellulases do exhibit a harshness reducing effect.

Due to the fact that the pH activity optimum varies considerably from one type of cellulase to another, two different activity determination methods are used in relation to the invention. The CMC cellulase activity unit (CMCU) is defined in the publication AF 187/3 (pH 7.0). The NOVO cellulase activity unit (NCU) is defined in the publication AF 187.2/1-GB (pH 4.8). Both publications are available on request from NOVO Industri A/S, Novo Alle, 2880 Bagsvaerd, Denmark.

In a preferred embodiment of the agent according to the invention the cellulase is Humicola insolens cellulase. This cellulase is commercially available, and furthermore exhibits a pH optimum sufficiently alkaline to be compatible with a detergent containing washing solution.

In a preferred embodiment of the agent according to the invention the cellulase is Sporotrichum pulverulentum cellulase. This cellulase exhibits a slightly acid pH optimum and is therefore well suited for treatment in a slightly acid aqueous medium with acid auxiliary treatment agents.

In a preferred embodiment of the agent according to the invention the cellulase is Fusarium oxysporum cellulase. This cellulase exhibits a pH optimum sufficiently alkaline to be compatible with a detergent containing washing solution.

In a preferred embodiment of the agent according to the invention the cellulase is Trichoderma reesei cellulase. This cellulase is commercially available, and furthermore, it exhibits a slightly acid pH optimun and is therefore well suited for treatment in a slightly acid aqueous medium with acid auxiliary treatment agents.

In a preferred embodiment of the agent according to the invention the agent contains a cellulase activity corresponding to at least 5 CMC cellulase activity units per gram of agent. In this manner the colour clarification effect can be provided with a reasonable dosage rate of the agent.

In a preferred embodiment of the agent according to the invention the agent is a non-dusting granulate. This formulation is cheap and it can, if desired, easily be incorporated into a detergent.

In a preferred embodiment of the agent according to the invention the agent is contained in a tablet. This embodiment provides an exact and easily controllable dosage.

In a preferred embodiment of the agent according to the invention the agent is an aqueous liquid, preferably with a cellulase stabilizing agent. This embodiment does not exhibit any dust hazard and is instantly distributed in the aqueous treating medium.

In a preferred embodiment of the agent according to the invention the agent is part of a detergent. If the agent according to the invention is used in just a small dosage simultaneously with each wash, the greyish appearance of the fabric will never develop.

In a preferred embodiment of the agent according to the invention the agent is contained in a bag of a material which disintegrates at a temperature of at least 40°C or is soluble in the treating liquid. This embodiment makes dosage extremely easy for the consumer.

Also the invention in its second aspect comprises a method for treatment of a fabric in order to provide colour clarification wherein the fabric is treated in an aqueous liquid together with the clarification agent according to the invention. It has to be emphasized that this method is not a washing process as the disordered fibres removed from the fabric are not dirt, but genuine constituents of the fabric. This treatment can be a soaking without movement of the treating liquid, or a treatment with simultaneous stirring or other dynamic influence exerted on the fabric in the treating liquid.

In a preferred embodiment of the method according to the invention the pH value and the temperature of the aqueous medium is favourable for the activity and stability of the cellulase, and the treatment time is sufficient for at least a reasonable degree of cellulolytic action. In this manner an acceptable colour clarification effect will be obtained.

In a preferred embodiment of the method according to the invention the pH value and temperature of the aqueous medium and the treatment time are selected with a view of obtaining a maximum or substantially maximum cellulolytic action. In this way an excellent colour clarification effect will be obtained.

In a preferred embodiment of the method according to the invention the cellulolytic activity of the aqueous medium is above 250 CMC cellulase activity units/l of aqueous medium. A cellulolytic activity below 250 CMC cellulase activity units/l of aqueous medium normally will not provide an acceptable colour clarification effect.

The cellulase preparations used in the examples as the active constituent of the clarification agent according to the invention were produced microbially in a manner known per se. The preparations were produced in pilot plant scale. Reference is made to the following table, which shows the main data for the

cellulase preparations with a reference No. for each preparation. For easier identification reference is made to the preparation Nos. in the examples.

Family	Microorganism	pH optimum	Cellulase activity CMCU/g	Cellulase activity NCU/g	Preparation No.	Example No.
Ascomycetes	Humicola insolens	6.5	14,760		1	1,2,3,4
	Myceliophthora thermophila	6.0	10,783		2	4
	Fusarium oxysporum		6,350		3	4
	Trichoderma reesei	4.8		1,680	4	5
Basidiomycetes	Sporotrichum pulverulentum			6,000	5	5
	Irpex lacteus			29,000	6	5

The invention will be illustrated by the following examples.

Example 1

Two old dark grey cotton shirts were cut into two equal pieces. The two half shirts were treated twice under the following conditions:

An AEG-washing machine was used, vide the brochure LAVAMAT, BELLA 802, AEG H 245240335 from AEG, Roskildevej 8-10, 2620 Albertslund, Denmark.

The used programme was No. 2 (40°C) with tap water and cellulase with an activity of 600 CMCU/litre (cellulase originating from preparation No. 1). The other two half shirts were not treated at all.

The cellulase treated halves were much more attractive than the untreated, i.e. the greyish look was eliminated totally on the cellulase treated halves.

Example 2

An old dark blue cotton shirt was cut into four pieces, which were treated under the following conditions:

A Terg-O-tometer test washing machine was used. The Terg-O-tometer test washing machine is described in Jay C. Harris, Detergency evaluation and testing, Interscience Publishers Ltd., 1954, page 60 - 61.

The test was carried out at 40°C and 100 rpm for 30 minutes. The cellulase used in this example was preparation No. 1. The cellulase dosages were 0, 250, 500, 1000 CMCU/litre. In all cases 3.5 g/l KH_2PO_4 and 7.3 g/l Na_2HPO_4 $2H_2O$ was added in order to obtain a pH value of 7 in the solutions.

The swatches were compared by a test panel and by measurement of light reflection respectively. The reflection measurements were carried out at 460 nm with an ELREPHO 2000 spectrophotometer from Datacolor S.A., Brandbachstrasse 10, CH-8305 Dietlikon.

The light reflection, %R (full drawn lines) versus cellulase activity in CMCU/l is shown in figure 1. The greyish shade of the untreated swatches causes a higher light reflection. The effect of cellulase on the grey shade appears clearly from fig. 1.

The test panel consisted of six persons, each ranking the swatches according to the visual appearance. The most attractive was assigned a score of 1, the second best a score of 2 and so on. The total score for each swatch was calculated by adding the scores given by each member of the panel. The result of the panel evaluation shown in fig. 1 (dotted lines) matches the light reflection well.

Also in the following figures (fig. 2-7) the full drawn lines represent light reflections, and the dotted lines panel evaluations.

Example 3

One black cotton shirt and one black 85% cotton/15% viscose shirt were cut into two equal pieces and washed 12 times under the following conditions:

A Miele washing machine was used; this washing machine is described in the brochure T-49052 from Miele Maskiner A/S, Gladsaxe Møllevej 15, 2860 Søborg, DK. The programme was "kulørt-vask" 40°C and simultaneously 5 g/l of the washing powder "Uldvask" from Blumøller was used. To the washing solution corresponding to one of the two pieces was added cellulase originating from preparation No 1 in an activity of 45 CMCU/litre.

After the 12 treatments the cellulase treated half had a bright colour and an attractive look, whereas the untreated half still had a greyish, unattractive appearance.

The colour clarification effect could be detected clearly after the third wash, and it was further developed during subsequent washes.

Example 4

An old dark blue cotton shirt was cut into swatches, which were treated under the following conditions:

A Terg-O-tometer test washing machine was used. The test was carried out at 40°C and 100 rpm for 30 minutes. The cellulases used in this example were preparations Nos. 1, 2, and 3 respectively. The cellulase dosages were 0, 1000, 2500 and 5000 CMCU/litre. In all cases 2.6 g/l of tris(hydroxymethyl)aminomethan and 1.2 g/l of maleic acid was added in oder to adjust the pH value to 7.1.

The swatches were compared as indicated in example 2. The results of the evaluation are shown in figure 2, 3, and 4 corresponding to preparation Nos. 1, 2, and 3, respectively. In all cases the panel evaluation and the light reflection matches well. In order to provide an impression of the scattering of the evaluation from one person to another in the panel reference is made to the following tables with values for panel evaluation and light reflection.

Preparation No. 1	Cellulase dosage (CMCU/l)			
Person No.	0	1000	2500	5000
1	4	3	1	2
2	4	3	1	2
3	4	3	1	2
4	4	3	1	2
5	4	3	1	2
6	4	3	2	1
Total score	24	18	7	11
%R at 460 nm	13.6	12.8	12.2	11.9

Preparation No. 2	Cellulase dosage (CMCU/l)			
Person No.	0	1000	2500	5000
1	4	3	2	1
2	4	3	1	2
3	4	3	1	2
4	4	3	2	1
5	4	3	2	1
6	4	3	1	2
Total score	24	18	9	9
%R at 460 nm	13.6	12.8	12.4	11.8

Preparation No. 3	Cellulase dosage (CMCU/l)			
Person No.	0	1000	2500	5000
1	4	3	2	1
2	4	3	2	1
3	4	2	3	1
4	4	3	2	1
5	4	3	2	1
6	4	3	1	2
Total score	24	17	12	7
%R at 460 nm	13.6	12.0	12.5	11.9

Example 5

An old dark blue cotton shirt was cut into swatches, which were treated under the following conditions:

A Terg-O-tometer test washing machine was used. The test was carried out at 40°C and 100 rpm for 30 minutes. The cellulases used in this example were preparations Nos. 4, 5, and 6 respectively. The cellulase dosages were 0, 1000, 2500, and 5000 NCU/litre for No. 4 and No. 5, and 0, 5000, 7500, and 10,000 NCU/litre for No. 6. In all cases 0.12 mole/l of CH_3COOH and 0.2 mole/l of NaOH were added in order to adjust the pH value to 4.8.

An untreated swatch was included in the evaluation.

The swatches were compared by a test panel (as described in Example 2) and by measurement of light reflection at 460 nm. The results of the panel evaluation and the light reflection measurements appear from Figs. 5, 6, and 7, corresponding to preparations 4, 5, and 6, respectively.

The horizontal lines on fig. 5, 6, and 7 correspond to the untreated swatch. Thus, it appears from fig. 5, 6, and 7 that the treatment without enzyme generates an even more greyish appearance.

The consistency of the panel evaluations in this example turned out to be quite similar to the already documented consistency of the panel evaluation in example 4.

Example 6

A used but clean cotton shirt with yellow, red, blue and green stripes was cut into two pieces of equal size. One piece was treated under the following conditons:

A MIELE W 761 washing machine was used, vide the brochure "Brugsanvisning for vaskemaskine W 761", Miele a/s, Erhvervsvej 2, 2600 Glostrup, Denmark.

The programme used was "Kort kulørtvask 60°C" i.e. one wash cycle of a duration of around 75 minutes, temperature increasing from 12°C to 60°C during the wash. The treating liquid was deionized water and cellulase with an activity of 6000 CMCU/litre (cellulase originating from preparation No. 1).

The other piece of the shirt was not treated at all.

The cellulase treated piece was much more attractive than the untreated, i.e. the greyish look was eliminated totally on the cellulase treated piece.

Figure 8 shows a black and white representation, showing the colour clarification effect. The left half was treated in accordance with foregoing Example 1 and the right half treated identically except that cellulase was omitted.

The features disclosed in the foregoing description, in the following claims and/or in the accompanying drawings may, both separately and in any combination thereof, be material for realising the invention in diverse forms thereof.

CLAIMS

1. Clarification agent for coloured fabrics, containing or consisting of cellulose based fibres, characterized by the fact that the clarification agent as an active constituent contains a cellulase.

2. Clarification agent according to claim 1, wherein the cellulase is Humicola insolens cellulase.

3. Clarification agent according to claim 1, wherein the cellulase is Sporotrichum pulverulentum cellulase.

4. Clarification agent according to claim 1, wherein the cellulase is Fusarium oxysporum cellulase.

5. Clarification agent according to claim 1, wherein the cellulase is Trichoderma reesei cellulase.

6. Clarification agent according to claims 1-5, wherein the agent contains a cellulase activity corresponding to at least 5 CMC cellulase activity units per gram of agent.

7. Clarification agent according to claims 1-6, wherein the agent is a non dusting granulate.

8. Clarification agent according to claim 1-6, wherein the agent is contained in a tablet.

9. Clarification agent according to claims 1-6, wherein the agent is an aqueous liquid, preferably with a cellulase stabilizing agent.

10. Clarification agent according to claims 1-9, wherein the agent is part of a detergent.

11. Clarification agent according to claims 1-9, wherein the agent is contained in a bag of a material which disintegrates at a temperature of at least 40°C or is soluble in the treating liquid.

12. Method for treatment of a fabric in order to provide colour clarification or to inhibit colour deterioration, wherein the fabric is treated in an aqueous liquid together with the clarification agent according to claims 1-11.

13. Method according to claim 12, wherein the pH value and the temperature of the aqueous medium is favourable for the activity and stability of the cellulase, and wherein the treatment time is sufficient for at least a reasonable degree of cellulolytic action.

14. Method according to claim 12-13, wherein the pH value and temperature of the aqueous medium and the treatment time are selected with a view of obtaining a maximum or substantially maximum cellulolytic action.

15. Method according to claims 12-14, wherein the cellulolytic activity of the aqueous medium is above 250 CMC cellulase activity units/l of aqueous medium.

16. The use of a clarification agent in accordance with any one of claims 1 to 11 to provide colour clarification or to inhibit colour deterioration of a fabric.

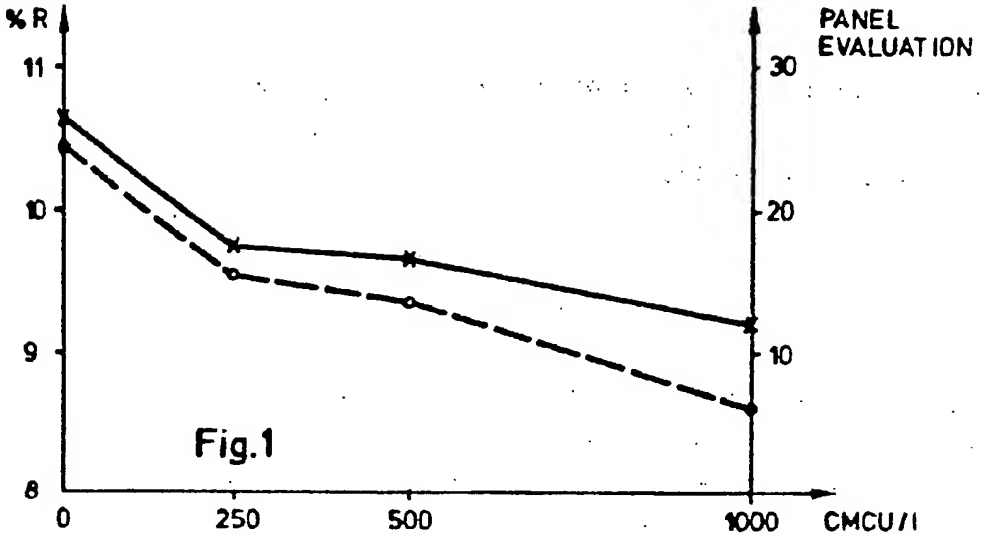

Fig.1

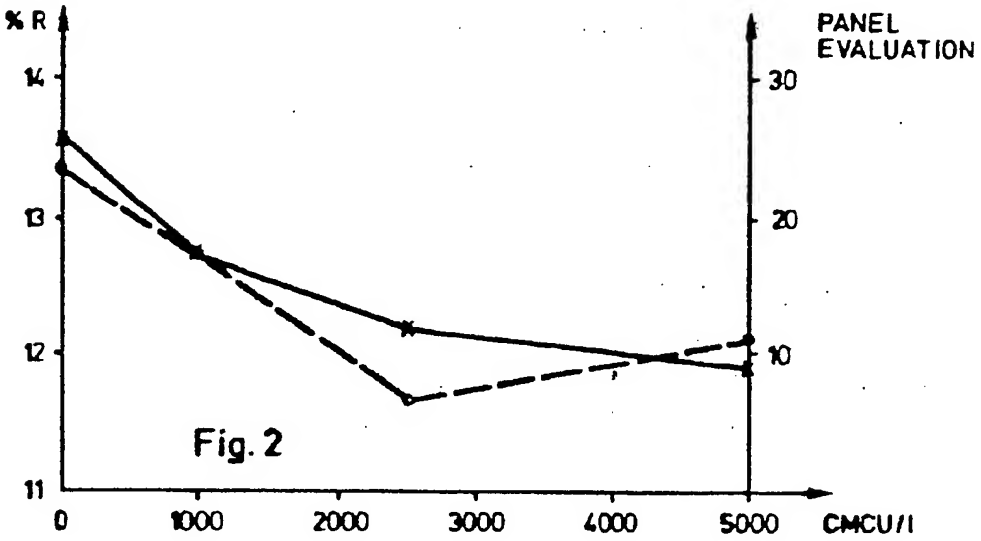

Fig. 2

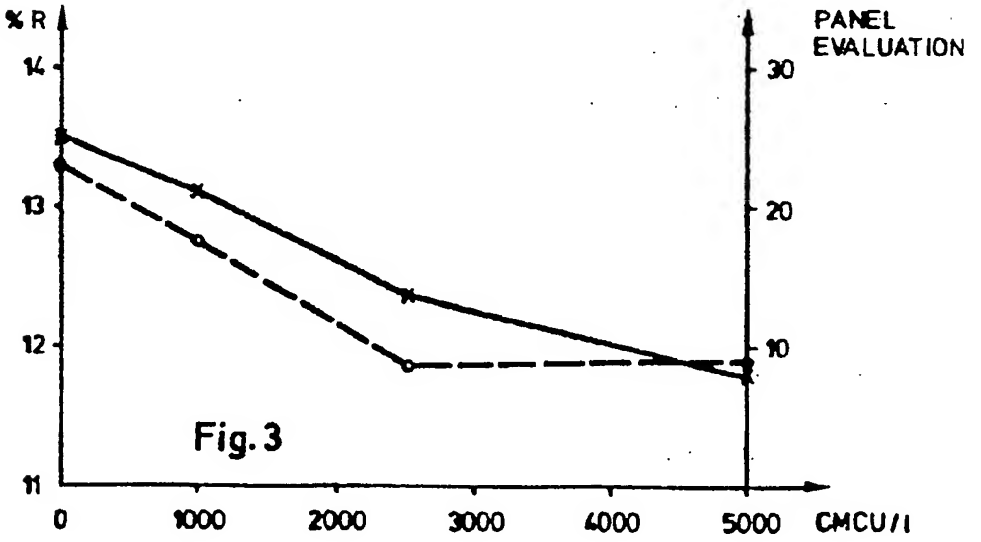

Fig. 3

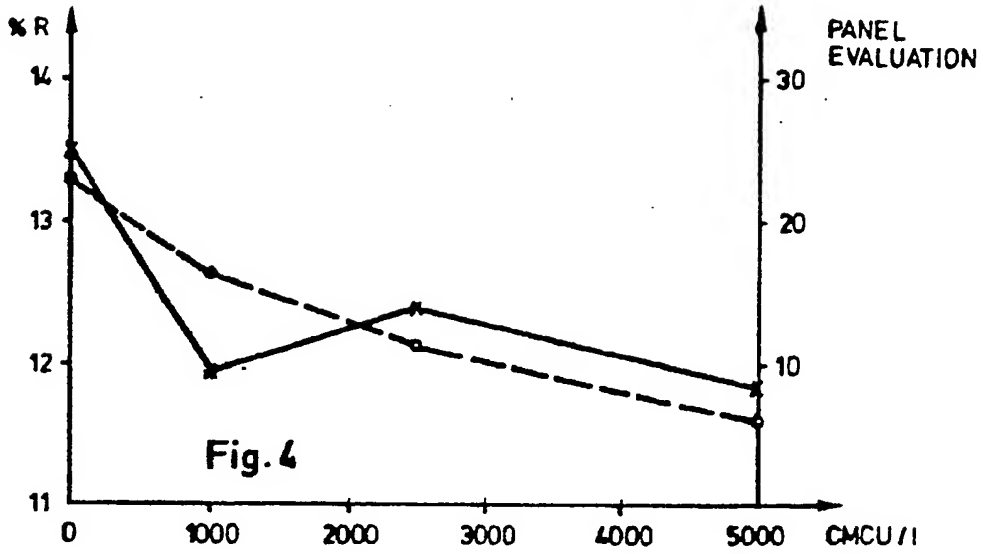

Fig. 4

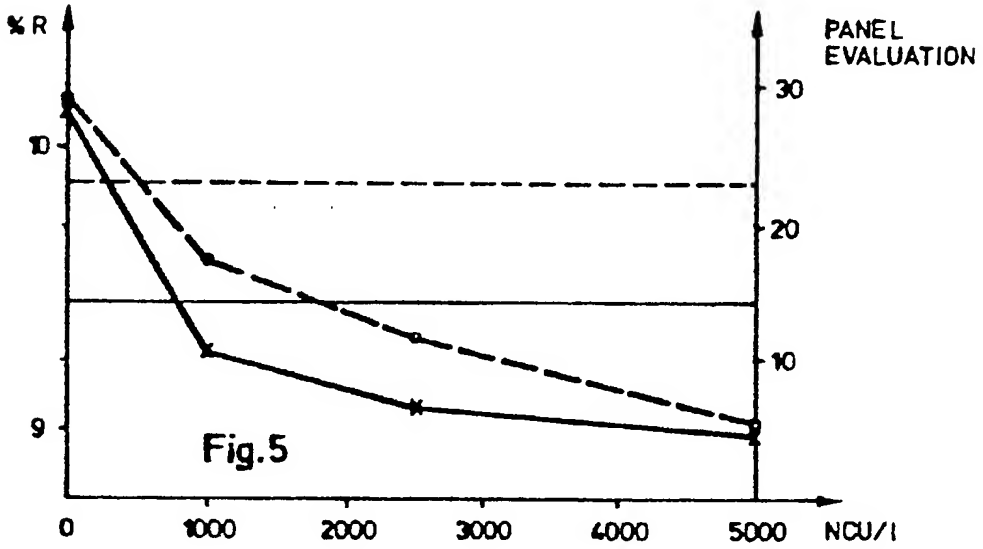

Fig. 5

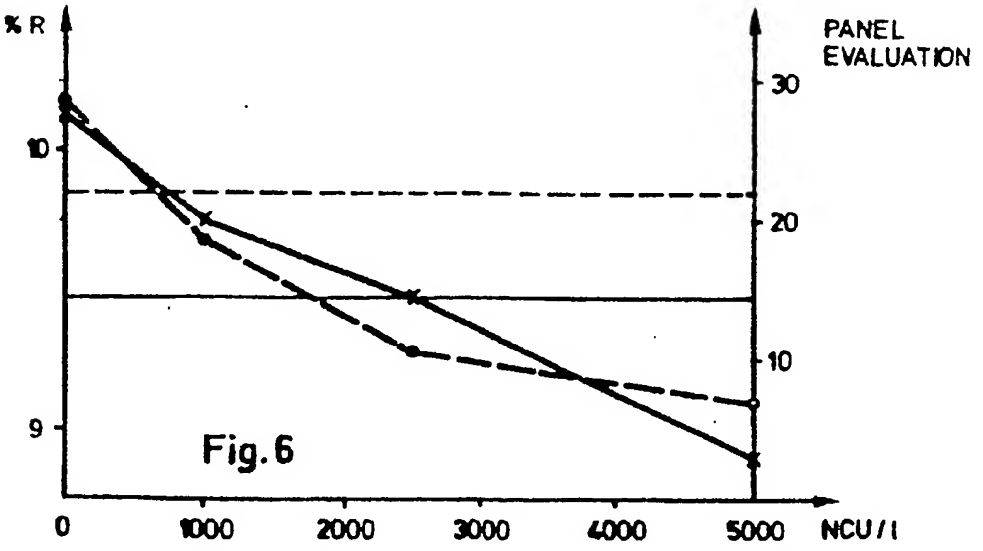

Fig. 6

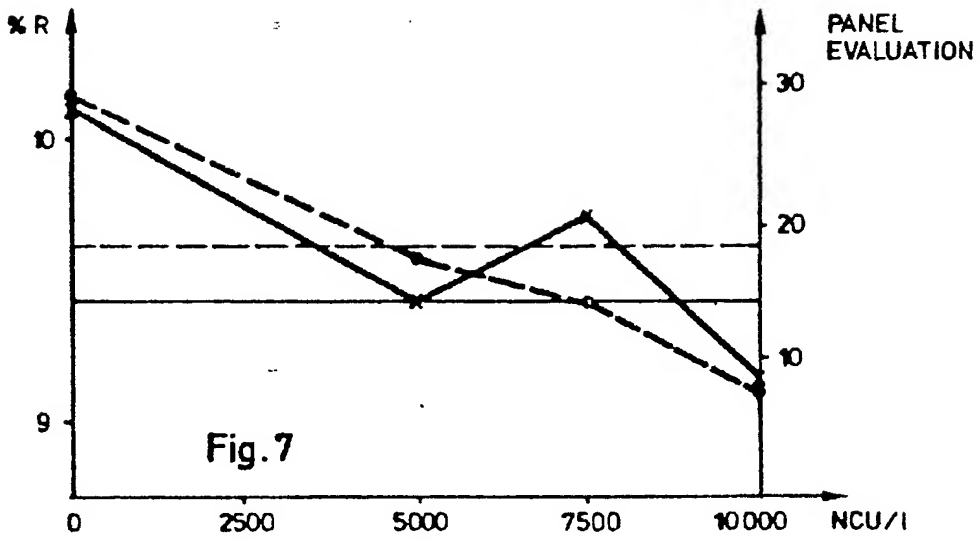

Fig.7

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Fig.8.